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IS 2741 (1989): Naphthol [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard
β-NAPHTHOL — SPECIFICATION
(Second Revision)

भारतीय मानक
β — नेफ्थाल — विशिष्टि
(दूसरा पुनरीक्षण)

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NEW DELHI 110002

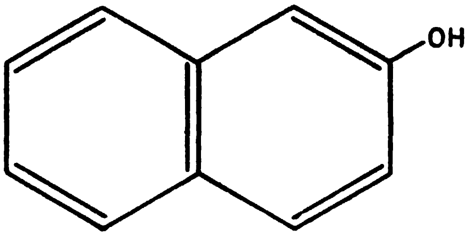
January 1990 **Price Group 2**

Dye Intermediates Sectional Committee, PCDC 11

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 27 January 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by Petroleum, Coal and Related Products Division Council.

β-Naphthol is an important raw material in the dyestuff industry. It is used in the manufacture of pigments and its derivatives are used in the manufacture of azo dyes. It is also known as 2-hydroxy-naphthalene and is represented by the following structural formula:



β-NAPHTHOL
(Molecular mass 144·2)
CAS Registry Number (135-19-3)

This standard was first published in 1964 and revised in 1973 in order to update the same in accordance with the quality of the material being produced at that time and also to introduce thin layer chromatographic method for estimation of α-naphthol content, which is the major impurity in β-naphthol. The alternate method for this purpose, namely, colorimetric method using sodium cupricyanide in alkaline solution was also substituted by a method using ferric chloride solution, chloroform or carbon disulphide.

The committee responsible for the preparation of this standard decided to revise it again in the light of the experience gained. In the present version (second revision), iodimetric method for assay has been introduced as an alternate method to coupling value method given in **13** of IS 5299 : 1969 'Methods of sampling and tests for dye intermediates'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 MARCH 2002
TO
IS 2741 : 1989 β-NAPHTHOL — SPECIFICATION

(Second Revison)

[*Page* 1, *Table* 1, *Sl No.* (ii)] — Substitute the following for the existing:

(1)	(2)	(3)	(4)	(5)
ii)	Insoluble in dilute sodium hydroxide solution, percent by mass, *Max*	0.2	when tested as per IS 5299 : 2001'	—

(PCD 11)

Reprography Unit, BIS, New Delhi, India

Indian Standard
β-NAPHTHOL — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for β-naphthol.

2 REFERENCES

IS No.	*Title*
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of dangerous goods: Part 1 Dangerous goods
IS 2552 : 1979	Specification for steel drums (galvanized and ungalvanized) (*second revision*)
IS 5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless to pale brown powder or scales. It shall be free from dust and other visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable paper bags, plywood boxes or drums conforming to IS 2552 : 1979.

4.2 Marking

The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer;

c) Batch number;

d) Tare, net and gross mass; and

e) The minimum cautionary notice worded as under:

> ACUTE IRRITANT. AVOID INHALATION AND CONTACT WITH SKIN. HANDLE WITH CARE. KEEP PROTECTED FROM HEAT. KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEEDS.

Table 1 Requirements for β-Naphthol

(*Clauses* 3.2, 5.3.1, 5.3.2 and 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Cl No. in IS 5299 : 1969	Annex
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, *Min*	120	7	—
ii)	Solubility in sodium hydroxide solution	To pass the test	10.2	—
iii)	Ash, percent by mass, *Max*	0.1	11.1	—
iv)	α-Naphthol content, percent by mass, *Max*	0.5	—	A
v)	Assay, percent by mass, *Min*	98.5	—	B

4.2.1 The containers shall also be marked with the symbol of 'Danger of Poisoning' (*see* IS 1260 (Part 1) : 1973].

5 SAMPLING

5.1 Preparation of Test Samples

The method of preparing representative samples of the material shall be as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Tests for assay and crystallizing point shall be conducted on each of the individual samples separately.

5.2.2 Tests for determination of the remaining characteristics, namely, α-naphthol content, ash, and solubility in sodium hydroxide solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay and crystallizing point if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **5.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in IS 5299 : 1979 and the Annexes as indicated in col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, Sl No. (iv)]

DETERMINATION OF α-NAPHTHOL CONTENT

A-1 METHOD 1

A-1.0 General

Dissolve the sample in alcohol, add aqueous ferric chloride solution and after mixing, extract with either chloroform or carbon disulphide. The presence of α-naphthol is indicated by the appearance of a blue-violet coloration in the solvent layer.

A-1.1 Reagents

A-1.1.1 *Standard Solution of α-Naphthol*

Dissolve 40 mg of chemically pure α-naphthol in alcohol and make up to 100 ml, one millilitre of this solution contains 0·4 mg of α-naphthol.

A-1.1.2 *Ferric Chloride Solution*, aqueous 5 percent (*m*/*v*).

A-1.1.3 *Chloroform*

A-1.1.4 *Carbon Disulphide*

A-1.2 Procedure

A-1.2.1 Dissolve 0·2 g of the material in 10 ml of alcohol. Add 0·5 ml of the ferric chloride solution and mix thoroughly. Add 5 ml each of water and chloroform or carbon disulphide, and shake.

A-1.2.2 To 0·200 g of α-naphthol-free β-naphthol, add 2·5 ml of standard solution of α-naphthol and 7·5 ml of alcohol, and treat with ferric chloride, water and organic solvent in the same way as given in **A-1.2.1**.

A-1.2.3 Compare the intensities of coloration of two solvent layers as obtained in **A-1.2.1** and **A-1.2.2**.

A-1.2.4 The limit test for α-naphthol is deemed to be complied with if the coloration intensity obtained in **A-1.2.1** is not greater than that obtained in **A-1.2.2**.

A-2 METHOD 2

A-2.0 General

The sample is chromatographed on a thin layer chromatographic plate to separate α-naphthol. The spots are made visible by subjecting the plates to the action of iodine fumes.

A-2.1 Apparatus

A-2.1.1 *Thin Layer Chromatographic Plate*

Glass plate of 10 cm × 20 cm, coated uniformly with silica gel G in a thickness of 250 micron and activated at 110°C for 30 minutes.

A-2.1.2 *Micropipettes*, 10 µl capacity

A-2.1.3 *Developing Chamber*

Suitable rectangular glass jar which contains the developer and which is closed well with the lid for saturation.

A-2.1.4 *Iodine Desiccator*

A glass desiccator which is saturated with iodine vapours by placing inside the desiccator a small cistern containing slightly warmed iodine crystals.

A-2.2 Reagents

A-2.2.1 *α-Naphthol β- Naphthol*

Purify 10 g of the technical grade β-naphthol by recrystallizing twice in hot water. The recrystallized product is filtered and dried at 80°C to constant mass.

A-2.2.2 *Methyl Alcohol*

A-2.2.3 *Eluent*, a mixture of benzene and ethyl acetate (97 : 3).

A-2.2.4 *Iodine*

A-2.2.5 *α-Naphthol*

A-2.3 Procedue

A-2.3.1 Prepare standard solutions of β-naphthol containing known amount of α-naphthol in the following manner.

> Weigh accurately 0·5 g each of α-naphthol-free β-naphthol in three 100-ml dry volumetric flasks. Then add 1·5, 2, and 2·5 ml of 0·1 percent solution of α-naphthol in methyl alcohol to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flasks by adding 10 ml of methyl alcohol and shaking them. Then make the volume to mark using methyl alcohol. Thus, we have three solutions of β-naphthol containing 0·3, 0·4 and 0·5 percent of α-naphthol, respectively. In a fourth 100-ml flask, weigh 0·5 g of the sample under test and make up the volume to mark with methyl alcohol.

A-2.3.2 Using the micropipette, spot 10 µl each of all the four solutions on to the thin layer chromatography plate. Care should be taken to see that all the spots are in the same line. Allow to dry. Then place the plate in the solvent of the developing chamber in a vertical manner and close the chamber. Allow to run for 20 minutes.

A-2.3.3 Take out the plate after 20 minutes and dry the solvent completely. Then place the plate in the iodine desiccator and examine the top violet spots. Compare the intensity of the spots visually with those of the known standard. Report the α-naphthol content as that which is nearest in intensity to the standard. In case the colour intensity of the sample does not come in the range of the standard spots, repeat the whole procedure using different percentages of α-naphthol.

ANNEX B

[*Table* 1, Sl No. (v)]

ASSAY IN β-NAPHTHOL

B-0 GENERAL

Two methods for assay in β-naphthol have been prescribed, namely Method A and Method B. In case of dispute, Method A shall be taken as the referee method.

B-1 METHOD A

Determine assay in accordance with the method given in 13 of IS 5299 : 1969 using 4-nitrobenzene diazonium chloride solution under neutral conditions.

B-2 METHOD B

B-2.1 Reagents

B-2.1.1 *Sodium Hydroxide Solution*, 10 percent (*m*/*v*).

B-2.1.2 *Glacial Acetic Acid*

B-2.1.3 *Sodium Bicarbonate*

B-2.1.4 *Iodine Solution*, 0·1 N.

B-2.1.5 *Starch Solution*

B-2.1.6 *Sodium Thiosulphate Solution*, 0·1 N.

B-2.2 Procedure

Weigh accurately about 0·5 g of the material in a 1 000 ml conical flask and dissolve in 50 ml of 10 percent sodium hydroxide solution. Add about 400 ml distilled water and mix well. Add glacial acetic acid till an incipient precipitate is formed. Add 30 g of sodium bicarbonate to the solution and titrate against freshly standardized iodine solution using starch solution as an indicator which is added just before the end point. Add 5 ml more of standard iodine solution and back titrate excess iodine after 5 minutes with standard sodium thiosulphate solution (**B-2.1.6**).

B-2.3 Calculation

Assay, percent by mass

$$= \frac{(V_1 \times N_1) - (V_2 \times N_2) \times 7{\cdot}208}{M}$$

where

V_1 = titre volume of iodine solution;

V_2 = titre volume of sodium thiosulphate solution;

N_1 = normality of standard iodine solution;

N_2 = normality of standard sodium thiosulphate solution; and

M = mass of sample taken for the test; g.

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